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(54) NEW FLUORINE-CONTAINING COMPOUND AND METHOD FOR PRODUCING THE SAME

(57)Abstract:

PROBLEM TO BE SOLVED: To obtain a new fluorine-containing nitrile compound containing a fluorosulfonyl group and a cyano group, having a boiling point higher than a normal temperature and ready handleability and to provide a method for producing the same.

SOLUTION: This fluorine-containing nitrile compound is represented by general formula  $\text{FSO}_2\text{CF}_2\text{CF}_2\text{O}(\text{CF}(\text{CF}_3)\text{CF}_2\text{O})_n\text{CF}(\text{CF}_3)\text{CN}$  ( $n$  is 0, 1 or 2). This compound is obtained by selectively amidating a fluoroformyl group-containing compound and converting the resultant substance into a nitrile.

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## DETAILED DESCRIPTION

[Detailed Description of the Invention]

[0001]

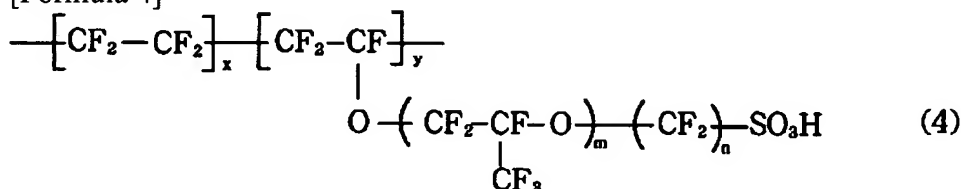
[Field of the Invention] This invention relates to the new fluorine-containing nitril compound which has a fluoro sulfonyl group and a cyano group, and its manufacture approach.

[0002]

[Description of the Prior Art] Conventionally, in the field of a fuel cell, the perfluorocarbon polymer (henceforth a perfluorocarbon-sulfonic-acid polymer) of the sulfonic group ( $-\text{SO}_3\text{H}$ ) content which has the structure of a formula (4) as the film and a proton conductor of a catalyst bed is mainly used.

[0003]

[Formula 4]



[0004] (As the inside of a formula, and x, comparatively, x:y is about 20:1-2:1 and m is an integer of y whose n is 1-12 in 0, 1, or 2.) [0005] This copolymer is  $\text{CF}_2=\text{CF}_2$ .  $\text{CF}_2=\text{CFO}$  [ $\text{CF}_2\text{CF}(\text{CF}_3)\text{O}$ ] Radical copolymerization of m ( $\text{CF}_2$ ) n  $\text{SO}_2\text{F}$  is carried out, and it is a fluoro sulfonyl group ( $-\text{SO}_2\text{F}$ ) to the end of a side chain. After obtaining the copolymer which it has and hydrolyzing this fluoro sulfonyl group by the base, it is compounded by carrying out the ion exchange with proton acid. Usually, the polymer of m= 1 and n= 2 is used.

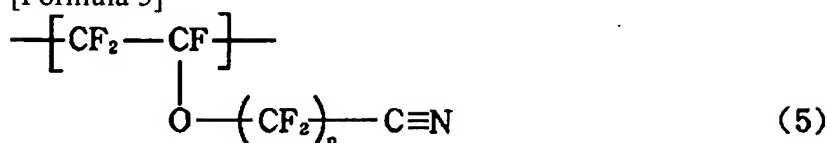
[0006] On the structure, since the above-mentioned perfluorocarbon-sulfonic-acid polymer did not contain any hydrogen, it is physical very [ chemically and ] stable, and can operate a fuel cell at comparatively high temperature, and its cell engine performance improved sharply. A deer is carried out, and as for a fuel cell, operating in an elevated temperature further is desirable in order to reduce the catalyst poisoning by the carbon monoxide contained in the hydrogen gas of a fuel. However, since this is a polymer non-constructing a bridge although the configuration is held with the par FURUO carbon chain when the above-mentioned polymer is formed in the film etc., the reinforcement and endurance in an elevated temperature have the technical problem that there is nothing, if not necessarily enough.

[0007] Although it was thought suitable to use fundamentally the perfluorocarbon-sulfonic-acid polymer over which the bridge was constructed in order to conquer the above-mentioned technical problem, the available bridge formation perfluorocarbon-sulfonic-acid polymer was not known industrially conventionally.

[0008] On the other hand, as a perfluorocarbon polymer which can construct a bridge, it is a side chain. - It is CN. The polymer which has a radical is well-known (see JP,6-340710,A etc.). formula which specifically has the following repeat unit (5) \*\* (6) The perfluorocarbon polymer expressed is illustrated.

[0009]

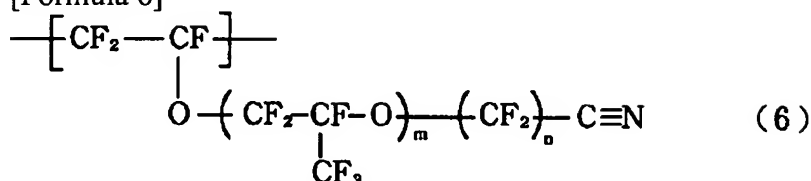
[Formula 5]



(The inside of a formula, integer of n=1-15)

[0010]

[Formula 6]

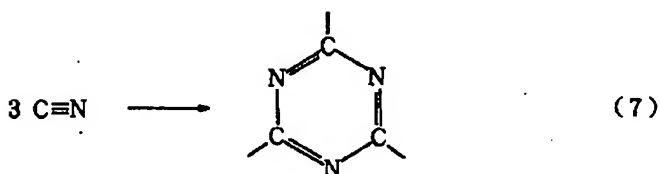


(The inside of a formula, integer of m= 1 and 2 n=1-4)

[0011] Inside of the compound expressed with the above-mentioned formula - CN It is a formula, using a radical as a catalyst for tetra-phenyl tin, triphenyltin hydroxide, Zn (C7 F15CO2)2, Co (C7 F15CO2)2, 1-perfluoro octane sulfonic acid, 2-ethylhexylamine, an aniline, etc. (7) A bridge formation perfluorocarbon polymer is formed by making a triazine ring generate by 3 quantification reactions.

[0012]

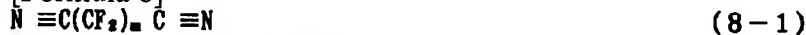
[Formula 7]



[0013] They are two pieces to intramolecular further again. - CN A bridge formation perfluorocarbon polymer can be obtained also by performing the reaction same about the perfluorocarbon compound which has a radical (for example, United States patent No. 3,317,484). They are two pieces to this intramolecular. - CN The compound concretely shown in following type (8-1) - (8-5) as a perfluorocarbon compound which has a radical is illustrated, and especially the boiling point can use a thing higher than near a room temperature suitably in these.

[0014]

[Formula 8]



(式中、n = 2 ~ 12 の整数)

(式中、n = 0 ~ 12 の整数、X=F 又は CF<sub>3</sub>)(式中、n = 2 ~ 12 の整数、X=F 又は CF<sub>3</sub>)(式中、n = 0 ~ 12 の整数、y = 0 ~ 100 の整数、X=F 又は CF<sub>3</sub>)(式中、n = 2 ~ 12 の整数、u+w = 0 ~ 100 の整数、X=F 又は CF<sub>3</sub>)

[0015] -SO<sub>2</sub>F [ therefore, ] A radical and -CN the perfluorocarbon compound which has both radicals -- compounding -- formula (5) \*\* (6) The time To a side chain - CN The perfluorocarbon polymer which has a radical, or formula (8-1) If the perfluoro compound which has two or more cyano groups like - (8-5) is mixed and crosslinking reaction is made to perform, it will be a fluoro sulfonyl group (-SO<sub>2</sub>F). It is thought that the bridge formation perfluorocarbon polymer which it has is obtained. Moreover, as described above, it is the -SO<sub>2</sub>F concerned. A radical is a sulfonic group (-SO<sub>3</sub>H) by hydrolysis and the ion exchange. It is changed.

[0016] Thus, -SO<sub>2</sub>F Radical - CN As a perfluorocarbon compound which has both radicals, FSO<sub>2</sub> CF<sub>2</sub> CN of a difluoromethane frame is well-known conventionally (Journal of Fluorine Chemistry, vol.84, and pp 135-139 (1997)). However, primarily, in this reference, the above-mentioned compound is compounded for the purpose of examining how physical properties, such as that NMR parameter, change, when the halogen (X) concerned of a halogenation difluoromethane fluoro sulfonyl [FSO<sub>2</sub> CF<sub>2</sub> X (X=Cl, Br, I)] is permuted by the cyano group. Moreover, the fluoro sulfonyl compound concerned is [ that it is only indicated that that cyano group reacts with a methanol, an aniline, a mercapto aniline, etc., and ], and, of course, neither a publication nor suggestion is in this reference about obtaining the fluoro sulfonyl group content bridge formation perfluorocarbon polymer which this invention plans and which is made to cause crosslinking reaction like the above using a cyano group, and can be used for the polyelectrolyte film of a fuel cell.

[0017]

[Problem(s) to be Solved by the Invention] A deer is carried out. this invention persons the compound of the above-mentioned difluoromethane frame For technical-problem achievement of using it as a start compound of the fluoro sulfonyl group content bridge formation perfluorocarbon polymer composition which can use this for the above-mentioned reference suitable for the polyelectrolyte film of the fuel cell in which a publication does not have even suggestion, either etc. It was dealt with extremely that the boiling point is too (24.0-24.5 degrees C) low, and tends to vaporize at the time of crosslinking reaction, and found out that there was a problem that a sex is bad. Then, it has two radicals, a fluoro sulfonyl group and a cyano group, in both ends, and the boiling point considered composition of the start compound of good bridge formation perfluorocarbon polymer composition higher than a room temperature of handling nature, and it resulted in this invention.

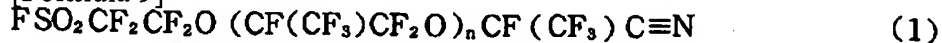
[0018] This invention has a fluoro sulfonyl group and a cyano group in this way, and aims to let the boiling point offer a new fluorine-containing compound sufficiently higher than ordinary temperature with easy handling, and the high manufacture approach of the selectivity.

[0019]

[Means for Solving the Problem] It will be a formula if this invention is followed. (1) New fluorine-containing nitril compound \*\* expressed is offered.

[0020]

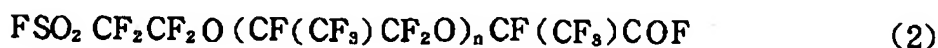
[Formula 9]



(Inside of a formula, n= 0, 1, or 2)

[0021] Moreover, it will be a formula if this invention is followed. (2) [0022]

[Formula 10]



(Inside of a formula, n= 0, 1, or 2)

[0023] The fluorine-containing compound and NH<sub>3</sub> which are come out of and expressed Formula characterized by performing dehydration after making it react and amidating alternatively the fluoro formyl group in the fluorine-containing compound concerned (1) Manufacture approach \*\* of a

fluorine-containing nitril compound expressed is offered.

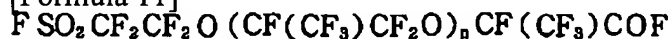
[0024]

[Embodiment of the Invention] Hereafter, this invention is explained to a detail.

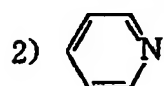
[0025] the fluorine-containing nitril compound of this invention -- for example, the following schemes -- following -- a carboxylic-acid full ora -- the id -- it is compoundable by two steps of reactions (9), and (10) from a derivative.

[0026]

[Formula 11]



1)  $(\text{CF}_3\text{CO})_2\text{O}$

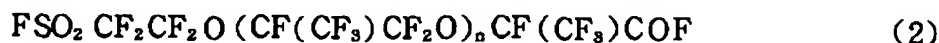


(n has among a formula the semantics which formula (1) already defined.)

[0027] first, the carboxylic-acid full ora whose reaction (9) of the preceding paragraph is acid halide -- the id -- it is the alternative amidation reaction of the fluoro formyl group of a derivative. Setting to this invention, the amidation concerned is a formula (2) concretely.

[0028]

[Formula 12]



(n has the already defined semantics among a formula.)

[0029] It is the fluorine-containing compound (carboxylic-acid full ora id derivative) come out of and expressed in an aprotic solvent and under low temperature  $\text{NH}_2$  You make it contact and it is carried out by amidating only a fluoro formyl group alternatively.

[0030] Especially as equipment for carrying out this amidation reaction, although it does not limit, it is an agitator, a thermometer, heating/cooling system, a temperature controller, and  $\text{NH}_3$ . The reaction container of the mixing vessel mold equipped with cooling dropping equipment or a weight or volumetric or counting feeder is desirable. Moreover, it can also consider as successive reaction actuation with the reaction container of a juxtaductal type.

[0031]  $\text{NH}_3$  Formula (2) As for the temperature to which a compound is contacted, it is desirable preferably that it is  $-80^\circ \text{C}$  -  $-30^\circ \text{C}$  low temperature  $-100^\circ \text{C}$  -  $30^\circ \text{C}$  in order to raise the selectivity of the amidation about a fluoro formyl group. At temperature lower than this, a reaction becomes slow and actuation of returning temperature to the above-mentioned range is needed. If temperature becomes higher than this, since it becomes impossible to disregard side reaction and the selectivity of amidation falls, it is not desirable.

[0032] As an aprotic solvent used for a reaction, diethylether, methyl-t-butyl ether, A tetrahydrofuran, dioxane, mono-glyme, a jig lime, a Torigu lime, Ether;2H, such as tetraglyme, methyl ether (perfluoro butyl), and ethyl ether (perfluoro butyl), a 3H-perfluoro pentane, A 1H-perfluoro hexane, a 1H-perfluoro

octane, a 1H-perfluoro decane, 1H and 4H-perfluoro butane, 1H, 1H, 1H and 2H, a 2H-perfluoro hexane, 1H, 1H, 1H, 2H, a 2H-perfluoro octane, 1H, 1H, 1H and 2H, a 2H-perfluoro decane, 3H and 4H-perfluoro (2-methyl pentane), 2H, and 3H-perfluoro (2-methyl pentane), 1 and 1-dichloro - 2, 2, 3, 3, and 3-pentafluoro propane, 1, 3-dichloro - 1, 1, 2, 2, a 3-pentafluoro propane, Fluoroalkanes or chloro fluoroalkanes, such as 1 and 1-dichloro-1-fluoro ethane, perfluoro (2-butyl tetrahydrofuran), 1,1,2-trichloro-1,2,2-trifluoroethane, and a perfluoro octane, are used suitably. In addition, halogenation aromatic hydrocarbon, such as aromatic hydrocarbon; fluorobenzenes, such as hydrocarbon; benzene, such as a hexane, a heptane, and an octane, toluene, and a xylene, a bromobenzene, a chlorobenzene, and a dichlorobenzene; although dimethylformamide, dimethylacetamide, dimethyl sulfoxide, hexamethylphosphoramide, a sulfolane, a tetramethyl sulfone, chloroform, a methylene chloride, a carbon tetrachloride, etc. can be used suitably, these are only instantiation and are not limited to this.

[0033] NH<sub>3</sub> the amount used -- formula (2) the carboxylic-acid full ora expressed -- the id -- a derivative -- receiving -- a 0.8 to 1.5 time mol -- it is a 1.0 to 1.2 time mol preferably. It is a formula when the amidation reaction of a fluoro sulfonyl group will advance with the amidation reaction of a fluoro formyl group (-SO<sub>2</sub> F->-SO<sub>2</sub> NH<sub>2</sub>), the selectivity of amidation of a fluoro formyl group will fall, if there are not much too many use mole ratios, and there are not much too few mole ratios. (9) The invert ratio to the amidation concerned falls.

[0034] NH<sub>3</sub> used for a reaction although you may also teach the whole quantity to a reaction container by package -- reaction temperature -- the above -- controlling to become a desirable temperature requirement, cooling dropping equipment or a metering pump is used, and adding or continuation adding serially is also suitably performed to the system of reaction. Moreover, when using a juxtaductal type reaction container, it is NH<sub>3</sub> to each part of a coil. A feed hopper is prepared and it is NH<sub>3</sub> from this feed hopper. By carrying out division installation, a reaction rate is also controllable.

[0035] Reaction time (mean flow-time [ in / in the case of a successive reaction tub / a reaction vessel ]) is reaction temperature and NH<sub>3</sub>. Although it may change with a mole ratio, the amount of use nonprotic solvents (namely, concentration of a reaction solute), etc., it is usually about 1 - 24 hours preferably for 0.5 to 30 hours.

[0036] a batch process and continuous system -- in order to complete a reaction in the case of which, you may pass through the process which carries out a temperature up even to ordinary temperature gradually.

[0037] formula (9) in order to make a fluoro formyl group amidate alternatively in an amidation reaction -- the concentration of a fluorine-containing compound -- NH<sub>3</sub> voice with desirable making it react in the superfluous condition to concentration -- it is one [ like ]. For that, the whole quantity of a fluorine-containing compound is taught to the reaction container, and it is NH<sub>3</sub>. It is also a desirable mode to carry out division supply with means, such as dropping, into the fluorine-containing compound concerned.

[0038] In addition, formula which is starting material in this invention (2) A compound is compoundable by making fluoro sulfonyl perfluoro ASECHIRUFURUORAI DO and hexafluoropropylene epoxide react as indicated by British JP,1,034,196,B.

[0039] Moreover, a latter reaction (10) is a nitril-ized reaction, makes the carboxylic amide generated by the formula (9) in the solvent which aprotic dried react with a dehydrating agent, and makes nitril generate.

[0040] Especially as equipment for carrying out nitril-ization, although it does not limit, the reaction container of the mixing vessel mold equipped with an agitator, a thermometer, heating/cooling system, a temperature controller, dropping equipment, or a weight or volumetric or counting feeder is desirable. Moreover, it can also consider as successive reaction actuation with the reaction container of a juxtaductal type.

[0041] The range of -30 degrees C - 100 degrees C of reaction temperature of nitril-izing is -20 degrees C - 60 degrees C preferably. At low temperature, a reaction rate falls not much from this, and when temperature is not much high, it becomes impossible to disregard side reaction from this.

[0042] A nonprotic solvent, especially the so-called bipolar nonprotic solvent are desirable like the case

of the above-mentioned amidation as a solvent in the case of carrying out nitril-ization. For example, a formula (11) [0043]

R-(CO)-NR'R" (R is the alkyl group of H or carbon numbers 1-6 among a formula (11), and R' and R" are the alkyl groups of carbon numbers 1-6 independently, respectively.) Moreover, R It connects with either R' or R", and cyclic structure may be formed, R' and R" may also be connected, and cyclic structure may be formed. The solvent of the amide system expressed is mentioned as a desirable thing. [0044] Formula (11) It sets and is R still more preferably. H Or it is a methyl group, and R' and R" are the same cases where they are a methyl group or an ethyl group, although [ both ] you may differ but. [0045] As an example of this desirable solvent, N and N-dimethylformamide, N, and N-dimethylacetamide and N-methyl pyrrolidone are mentioned.

[0046] In addition to this as a desirable bipolar nonprotic solvent, dimethyl sulfoxide, a sulfolane, propylene carbonate, ethylene carbonate, gamma-butyrolactone, tetramethylurea, etc. are mentioned. Furthermore, the aprotic solvent indicated as usable can also be used in the amidation reaction mentioned above besides this.

[0047] As a dehydrating agent, the usual dehydrating agent is usable, for example, a phosphorus pentaoxide, an acetic anhydride, a trifluoroacetic acid anhydride, phthalic anhydride, phosphorus oxychloride, a thionyl chloride, etc. are mentioned.

[0048] In addition, in this invention, it is desirable to make the basic compound which carries out the trap of the acid which carries out a byproduction live together with a dehydrating agent in the nitril-ized reaction which uses the dehydrating agent which serves as an acid after dehydration.

[0049] As a desirable basic compound, it is the amine which H atom has not combined with N directly. A trimethylamine, triethylamine, tree n propylamine, The 3rd class fatty amines, such as tree i-propylamine, tree n butylamine, and triamylamine; 1, 5-diazabicyclo [4, 3, 0] -5-nonene, 1 and 5-diazabicyclo [4, 3, 0] non -- alicyclic amines [ , such as -5-en, ]; -- aromatic amine [ , such as dimethylaniline, ]; -- a pyridine -- A permutation or unsubstituted pyridines, such as methylpyridine (picoline), lutidine (lutidine), an ethyl pyridine, and a trimethyl pyridine (collidine), are mentioned.

[0050] In the above-mentioned scheme, it is the example which is reacting at 0 degree C by using a pyridine as a basic compound.

[0051] Especially the desirable combination of the above-mentioned dehydrating agent and a basic compound is mentioned as what has a desirable combination of a trifluoroacetic acid anhydride and a pyridine, for example, although it does not limit.

[0052] Although the amount of the dehydrating agent used may change with the class of dehydrating agent, the amount of the amide which should dehydrate, etc., 1.0-5.0 mols per one mol of carbamoyl groups, it is about 1.0-3.0 mols preferably, for example, when using a trifluoroacetic acid anhydride, it is 1.0-2.0 mols preferably 1.0-2.5 mols per one mol of carbamoyl groups.

[0053] Moreover, although the amount of the basic compound used may change with the class of the basic compound concerned, the amount of a dehydrating agent, etc. Usually, 1.0-4.0 mols are about 1.0-3.0 mols preferably to one mol of dehydrating agents. For example, when using a trifluoroacetic acid anhydride as a dehydrating agent and using pyridines and a tertiary amine compound as a basic compound, the 1.0-2.5 mols of the amount of the amine compound used concerned are 1.0-2.0 mols preferably to the one mol of the trifluoroacetic acid anhydrides concerned.

[0054] although a dehydrating agent (and basic compound) may also teach the whole quantity to a reaction container by package like the case of an amidation reaction -- dropping equipment or a metering pump -- using it -- the system of reaction -- serially -- addition or continuation addition -- carrying out -- making -- reaction temperature -- the above -- it is desirable to advance a reaction, controlling to become a desirable temperature requirement. Moreover, when using a juxtaductal type reaction container and carrying out a nitril-ized reaction continuously, temperature control can also be performed by preparing feed hoppers, such as a dehydrating agent, in each part of a coil, and carrying out division installation of the dehydrating agent etc. from this feed hopper like the case of an amidation reaction.

[0055] Although the reaction time (mean flow-time [ in / in the case of a successive reaction tub / a reaction vessel ]) of nitril-izing may change with reaction temperature, a dehydrating-agent mole ratio,

the amount of use nonprotic solvents (namely, concentration of a reaction solute), etc., it is usually about 1 - 24 hours preferably for 0.5 to 30 hours.

[0056] The carbamoyl group of an amide compound is changed into a cyano group by the above actuation by the reaction (formula (10)) with dehydrating agents, such as a trifluoroacetic acid anhydride and a phosphorus pentaoxide.

[0057] Formula (10) When the reaction which nitril generates from a fluoro amide is performed by the approach of this invention, the fluoro nitril to generate is separated as a phase which most becomes from the fluoro nitril concerned with the solvent phase containing the reaction residue with a dehydrating agent and a dehydrating agent. Subsequently, it is possible by dissociating from a solvent phase and rinsing the fluoro nitril content phase concerned with a liquid-liquid separation means, to obtain fluoro nitril by high yield. Usually, finally the fluoro nitril of a high grade is isolated by carrying out distillation purification of the fluoro nitril content phase.

[0058] If the approach of this invention is followed, the yield of the fluoro nitril from a fluoro amide will be very high, will be at least 50% or more, and will usually exceed 70%.

[0059] In addition, it can mix with a dehydrating agent and non-solvents, such as a phosphorus pentaoxide, besides the approach of performing in the nonprotic solvent of the above-mentioned publication, and a nitril-ized reaction can also adopt the approach of heating at 100-300 degrees C.

[0060] By the above reaction, it is a formula. (1) The fluorine-containing nitril compound of this invention expressed can be obtained with sufficient selectivity. In addition, formula (1) It is the formula which it sets, and n is 0, 1, or 2, and is a start raw material. (2) According to n, the number of n of a fluorine-containing nitril compound also becomes settled. Formula (3) The case of n= 0 is shown.

[0061]

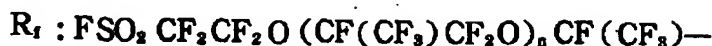
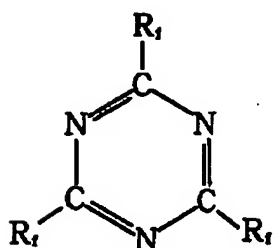
[Formula 13]



[0062] Formula which has the fluoro sulfonyl group and cyano group of this invention in both ends (1) A fluorine-containing nitril compound is a formula by 3 quantification reactions of the cyano group of an end. (12) It is possible to change into the triazine ring content compound expressed.

[0063]

[Formula 14]



(12)

(n expresses among a formula the semantics which formula (1) already defined.)

[0064] A triazine ring formation reaction can be carried out by the approach that a publication is well-known in itself, to JP,47-17793,B or JP,6-340710,A.

[0065] Namely, a bismuth, copper, lead, barium, cadmium, tin, a thallium, Metal catalysts, such as cadmium and an indium, tetra-phenyl tin, triphenyltin hydroxide, Under existence of metal oxide catalysts, such as Zn (C7 F15CO2)2, 1-perfluoro octane sulfonic acid, and a silver oxide In solvents, such as a non-solvent or a carbon tetrachloride, a cyclohexanone, dioxane, dimethyl sulfoxide, fluoride



benzene, a nitrobenzene, mono-glyme, a jig lime, and a TORIGU lime 0-400 degrees C, it is about 25-250 degrees C in temperature preferably, and is a formula. (1) What is necessary is just to make 3 quantification reactions (triazine ring formation reaction) of a fluorine-containing nitril compound perform for about 1 to 200 hours. in addition -- usually -- a catalyst -- formula (1) 0.01 - 10 mass % extent use of a nitril compound -- it carries out.

[0066] Formula (12) The triazine compound expressed is useful as intermediate field of a low-molecular electrolyte or a polymer electrolyte. That is, although the compound concerned has three fluoro sulfonyl groups in 1 molecule, this fluoro sulfonyl group can be easily changed into a sulfonic group or its salt by hydrolysis.

[0067] Moreover, formula of this invention (1) A fluorine-containing nitril compound is convertible for the crosslinked polymer containing a fluoro sulfonyl group (they are a sulfonic group or its salt by hydrolyzing further) by the above mentioned approach.

[0068] If the low-molecular electrolyte or polymer electrolyte of the triazine ring content which has the sulfonic group which are obtained by carrying out like the above is used as a lithium salt mold ( $-\text{SO}_3 \text{Li}$ ), it can be used as an electrolyte for lithium rechargeable batteries, and can be suitably used with an acid type ( $-\text{SO}_3 \text{H}$ ) as the film of a solid-state polyelectrolyte mold fuel cell, or a polymer for electrode layers.

[0069]

[Example] Hereafter, an example explains this invention concretely.

[0070] [Example 1]

(1) 346g (one mol) was inserted [ diethylether ] in the 4 opening flask mold reaction container of 3L equipped with the agitator, the thermometer, and the Dewar bottle mold dry ice condensator for 1450ml and fluoro formyl compound  $\text{FSO}_2 \text{CF}_2 \text{CF}_2 \text{OCF}(\text{CF}_3) \text{COF}$  as a nonprotic solvent, and it cooled at -70 degrees C after the nitrogen purge. while cooling  $\text{NH}_3$  17g (one mol) with a dry ice condensator there -- the bottom of churning -- liquefaction  $\text{NH}_3$  \*\*\*\*\* -- it was dropped.  $\text{NH}_3$  After completing dropping, it agitated for 0.5 hours and returned to the room temperature over 2 hours.

[0071] Then, pure water washed reaction rough liquid 3 times, and the separating funnel separated the organic phase. It is  $\text{FSO}_2 \text{CF}_2 \text{CF}_2 \text{OCF}(\text{CF}_3) \text{CONH}_2$  as a white solid-state by distilling off a solvent under reduced pressure, after magnesium sulfate performs moisture removal of an organic phase. Fluoro amide 303g (0.839 mols) expressed was obtained.

[0072] (2) Subsequently, fluoro amide  $\text{FSO}_2 \text{CF}_2 \text{CF}_2 \text{OCF}(\text{CF}_3) \text{CONH}_2$  equipped with the agitator, the thermometer, the dropping funnel, and the cooling pipe obtained by the 4 opening flask mold reaction container of 3L by dimethylformamide 1200g (1270ml) and the above as a nonprotic solvent 286g (0.834 mols) was inserted in, it considered as the dimethylformamide solution, and this was cooled at 0 degree C. 221g (1.05 mols) of anhydrous trifluoroacetic acid was slowly dropped there over 0.5 hours as a dehydrating agent. Pyridine 167g (2.11 mols) was further dropped slowly over 1 hour after dropping termination. Reaction temperature was maintained among 2-4 degrees C in the meantime.

[0073] After dropping termination, it agitated for 0.5 hours and 350g of pure water was added from the tap funnel, and it was left until it became a room temperature. The lower phase of the reaction rough liquid which carried out two-phase separation was isolated preparatively with the separating funnel, distillation was performed after pure-water washing 3 times, and 201g (0.583 mols, 70.3% of yield) of fluoro nitril  $\text{FSO}_2 \text{CF}_2 \text{CF}_2 \text{OCF}(\text{CF}_3) \text{CN}(\text{s})$  was obtained.

[0074] The analysis value of the fluoro nitril which is this fluorine-containing compound was as follows.

Boiling point 89 degrees C,  $^{19}\text{F}$ -NMR ( $\text{CD}_3$ ) (2  $\text{C}=\text{O}$  solvent and  $\text{CFCl}_3$  criteria,  $\delta$ ppm) 46.1 (1F, s), -79.8 (1F, dd), -82.8 (1F, d), -83.1 (3F, s), -111.7 (2F, s), -114.4 (1F, m)

[0075] Like the above, the fluorine-containing nitril compound of this invention is understood that the boiling point is sufficiently higher than ordinary temperature, and handling is easy.

[0076]

[Effect of the Invention] Formula which has the fluoro sulfonyl group and cyano group of this invention in both ends (1) The fluorine-containing nitril compound expressed is new, and useful as intermediate

field of the low-molecular electrolyte of triazine ring content, or a polymer electrolyte.

[0077] moreover -- if the approach of this invention is followed -- formula (2) amidating a fluoro formyl group with sufficient selectivity by using a compound as starting material, and nitril-izing further -- yield -- high -- formula (1) A fluorine-containing nitril compound can be obtained.

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[Translation done.]